Measuring Vapour Pressure with An Isoteniscope – A Hands-On Introduction to Thermodynamic Concepts

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ABSTRACT

Characterisation of the vapour pressure of a volatile liquid or azeotropic mixture, and its fluid phase diagram, can be achieved with an isoteniscope and an industrial grade
10 digital pressure sensor using the experimental method reported in this study. We describe vapour-pressure measurements of acetone and *n*-hexane and their azeotrope, and how the data can be used to calculate thermodynamic properties of the test liquids, such as the molar heat of vaporisation. This hands-on experience allows students to appreciate important thermodynamic concepts such as phase equilibrium, preparing
15 them for more-advanced studies of the subject.

ABSTRACT GRAPHIC

KEYWORDS

*First-Year Undergraduate / General, Chemical Engineering, Laboratory Instruction, Hands-
20 On Learning / Manipulatives, Laboratory Equipment / Apparatus, Thermodynamics, Phases/Phase Transitions/Diagrams*

25 INTRODUCTION

A key question in the design and operation of many processes involving fluids is “What is the state of the fluid?” Among the first things taught to undergraduate students of chemical engineering embarking on the study of thermodynamics is the need to continually ask themselves this question as part of their analysis of whatever
30 process they may be considering. In asking this question, we recognise the importance of understanding where and when within the process the fluid will be present as *liquid*, as *vapour*, or as a *mixture of (saturated) vapour and liquid in equilibrium* with each other (*VLE*). Examples of important industrial processes in which this knowledge is vital include: chemical separation processes such as distillation; heat engines and
35 refrigeration cycles that exploit the phase change as part of their function; cryogenic processes; fluid transport in pipelines due to the difference in the flow characteristics of single-phase and two-phase (or multi-phase) flow.

The answer to the question “what is the state of the fluid?” is obtained from knowledge of the fluid phase diagram, in which the position of the phase boundary
40 between the liquid and vapour phases is mapped out. This phase boundary is commonly known as the vapour-pressure (or saturation) curve. A typical example of a fluid phase diagram (plotted in the usual pressure, p , and temperature, T , space) is given in Figure 1. Clearly, the class of experiments through which such diagrams are determined is of fundamental importance.

Figure 1. A typical p - T fluid-phase diagram, illustrating the liquid and vapour regions, and the vapour-pressure curve.

The accepted standard test method for investigating the relationship between vapour pressure and temperature is detailed in ASTM standard D-2879-10, which was
50 first published in 1970 and recently revised in 2010¹. The ASTM standard describes the use of an isoteniscope to measure the vapour pressure of a pure liquid or liquid mixtures against an inert gas at a known pressure. The valid pressure range is between 133 Pa (1.0 Torr) and 101.3 kPa (760 Torr) and the temperature range is between 293 K
55 and 748 K¹.

The most common demonstration used for education purposes to date is that of Koubek^{2,3}, who employed a U-tube manometer to measure the vapour pressure of benzene with a static method, mainly to measure vapour pressure and to illustrate Raoult's Law. More recently, Pearson⁴ reported a simple demonstration using water
60 and aqueous salt solutions. In this experiment, comparison is made between de-ionised water and a salt solution in closed containers. The differences in the degree of condensation within the two containers highlights the differences in vapour pressure.⁴ An inexpensive method for vapour-pressure measurement has been proposed recently by DeMuro et al.⁵ Using this method, in which a bubble is trapped in a graduated
65 microtube prepared from a small pipette, the vapour pressures and enthalpies and

entropies of vaporisation of different organic solvents have been determined at different temperatures and were found to agree well with literature.

The use of an isoteniscope as a tool for measuring vapour pressure has been proposed before by Smith and Menzies⁶, and included in a teaching context by Steinbach⁷ and Sternberg⁸. Dannhauser⁹ proposed an experimental validation of the third law of thermodynamics for two different reactions, involving the isoteniscope as a tool for characterisation of vapour pressure. An experimental set-up on which most of the modern experiments are based was first proposed by Van Hecke¹⁰. Van Hecke used a complex isolation manifold involving four different valves to ensure that vapour and liquid phases are in equilibrium. Garlang, Nibler, and Shoemaker proposed using an isoteniscope for chemistry teaching purposes¹¹, while Halpern and McBane reported a much simpler and more modern design using a pressure transducer which required regular calibration¹².

Vapour pressure measurements using an isoteniscope have been used widely in teaching undergraduates experimental physical chemistry. Over the years methods have been refined to reduce complexity and enhance precision of the measurements. The experiment presented in this article builds on previous work by updating the design of the isoteniscope assembly and using an industrial grade absolute piezo transducer, which combines a MEMS (microelectromechanical)-based high precision piezo sensor with a metal sealed SS316 diaphragm and integrated electronics, and a digital pressure display. Our intent was to increase simplicity and robustness while maintaining the inherent elegance and accuracy of the classic technique. The combination of the high precision pressure transducer with a digital display means that students do not have to calibrate the sensor before each experiment, which saves time and avoids any calibration errors.

This experiment was specifically developed for first-year undergraduate students in chemical-engineering to obtain a “hands-on” appreciation of the VLE phenomenon by fusing traditional experimental techniques with modern laboratory technology. As laboratory-based teaching occupies significantly less space in a chemical-engineering curriculum than in a comparable chemistry programme, we
95 wanted to retain as many practical elements as possible to train essential fine-motor and manual handling skills while streamlining the technical procedure.

A further goal was to show how information about the phase boundary can be used to determine thermodynamic properties essential for engineering design
100 calculations. These include vapour pressures of the test fluids determined using the Clausius-Clapeyron equation¹³. This equation can be conveniently written in integrated form as:

$$\ln\left(\frac{p_{sat(2)}}{p_{sat(1)}}\right) = \frac{\Delta H_m^{vap}}{R} \left(\frac{1}{T_{sat(1)}} - \frac{1}{T_{sat(2)}} \right) \quad (1)$$

where p_{sat} and T_{sat} are the pressure and temperature at saturation, subscripts (1) and
105 (2) refer to the corresponding pressure / temperature pair at conditions (1) and (2), R is the universal gas constant, and ΔH_m^{vap} is the molar heat of vaporisation. Note that this equation can be easily extended to ideal and non-ideal binary mixtures, where ΔH_m^{vap} is taken to be the average molar heat of vaporisation of the mixture, weighted according to the mixture composition¹⁰.

MATERIALS

Chemicals

Reagent-grade acetone (CAS number: 67-64-1) and *n*-hexane (CAS number: 110-54-3) were obtained from VWR International Ltd. Lutterworth, UK and used
115 without further purification.

Equipment

(b)

Figure 2. (a) Schematic of the experimental set-up, and (b) photo of actual set-up.

140 Figure 3. Photograph of the isoteniscope (ruler units are in centimetres)

As shown in Figure 2 (a), the major components of the experimental set-up include an isoteniscope (GPE Scientific Ltd., Leighton buzzard, UK), a ballast tank (GPE Scientific Ltd., Leighton buzzard, UK), a diaphragm vacuum pump (ILMVAC GmbH, 145 Ilmenau, Germany), a piezo pressure transducer with digital pressure display (MKS Instruments, Inc., Andover, MA), two valves (GPE Scientific Ltd., Leighton Buzzard, UK), a glass cold trap (GPE Scientific Ltd., Leighton Buzzard, UK), and a water bath (Julabo UK Ltd., Peterborough, UK).

The isoteniscope itself (Figure 3) was designed at Imperial College London, and 150 custom made by GPE Scientific Ltd according to the technical drawing, which is provided in the supplementary information to enable easy reproduction of the instrument. The selection of the vacuum pump depends on the physical properties of the liquid/ liquid mixture to be investigated. For our purposes, the diaphragm vacuum pump, model number 412421, proved to be adequate. It can generate pressures less 155 than 60 mbar (as per DIN 28432), and is suitable for the temperature range of 10 to 60

°C. The piezo transducer, series 902B, provides absolute pressure measurement with a measurement range of 0.1 to 1000 Torr. The sensor has an accuracy of $\pm 1\%$ of reading, a reproducibility of $\pm 0.3\%$ and is supplied pre-calibrated by the manufacturer. This instrument is typically used in industries requiring high precision pressure measurements, e.g. semiconductor manufacturing, thin film coating, and aerospace, and does not require calibration by the end user. This saves time and allows students to focus on the main purpose of the experiment within a three-hour lab session, which is generating VLE data. Calibration of instruments is taught in a separate experiment in our laboratory and is out of scope of this article. The sensor is very robust and has proven itself since 2011 without any operational issues. A Julabo ED-5MB open heating batch circulator with transparent tank completes the assembly. It can readily control temperature in the range of 20 to 100 °C with set-point deviation of $\pm 0.03^\circ\text{C}$. All experiment components were installed as received.

The isoteniscope (Figure 3) is submerged in a water bath that can be heated in a controlled manner. It is connected with the ballast tank, which is opened to the atmosphere through valve 1 and connected to the vacuum pump through valve 2. The pressure sensor measures the pressure inside the ballast tank. When the liquid levels in the U-tube are horizontally balanced, the pressures on both sides are equal and hence the pressure indicated by the pressure sensor is the same as in the bulb of the isoteniscope. A cold trap (water-ice mixture in a Dewar flask) is in place between valve 2 and the vacuum pump to prevent any vapour of the test liquid entering the pump. Figure 2 (b) displays the complete experimental assembly on the laboratory bench. As can be seen, it is reasonably compact and from experience we can also claim that it is considerably robust: In almost five years of operation and handling by 120 first year students each year, so far only one isoteniscope had to be physically repaired because of glass breakage.

The question of “why use an isoteniscope when one can measure the vapour pressure using the direct method?” may spring to mind of the interested reader. The direct measurement of vapour pressure in a simple flask containing the test liquid is undoubtedly more convenient. However, the accuracy of such a method depends on the simultaneous temperature control of the vapour and liquid phases. Hence, the entire equipment that contains both phases has to be at the same temperature, so that a thermal equilibrium can be established. In a simple flask, the section which leads to the pressure sensor often lacks this temperature control because of its exposure to room temperature, which makes it difficult for the two phases to reach thermal equilibrium. As a result, this causes a systematic error in the measurement of vapour pressure. The use of an isoteniscope ensures that both vapour and liquid phases are always subject to the same temperature, as bulb and manometer section are submerged simultaneously. In addition, the user of an isoteniscope can easily see whether the vapour and liquid phases are at equilibrium by checking the liquid level in the manometer section, which is one of the key learning experiences we want to pass on to the students.

METHOD

The bulb of the isoteniscope is filled with the test liquid to approximately three-quarter height, so that no liquid flows into the U-tube when the isoteniscope is placed vertically. The isoteniscope is then connected to the ballast tank which is at atmospheric pressure (valves 1 and 2 are closed), before it is placed in the water bath, which is set to 25 °C.

Valve 2 is opened slowly to allow the vacuum pump to evacuate the air from the system. As the pressure inside the system decreases below the vapour pressure of the test liquid at 25 °C, the test liquid in the bulb of the isoteniscope starts boiling. The vacuum is maintained for another five minutes in order to remove as much air from the system as possible. Valve 2 is then closed and the vacuum pump is switched off. The

isoteniscope is tilted slightly so that some of the test liquid flows into the U-tube. Once
210 the bubbling liquid fills up the U-tube, the U-tube is placed vertically.

Valve 1 is opened slowly to allow air to enter the ballast tank in order to increase
its internal pressure. Boiling ceases and the liquid level on the ballast tank side of the
U-tube decreases slowly until it is level with the bulb-side liquid level. Valve 1 is then
closed. The pressure indicated by the pressure sensor is recorded, as is the temperature
215 of the water bath.

The water bath temperature is increased by five degree centigrade and valve 1 is
opened gradually to maintain the balanced liquid levels in the U-tube. Once the
temperature of the water bath stabilises and the liquid levels in the U-tube are at equal
height, valve 1 is closed. The pressure indicated by the pressure sensor is recorded with
220 the temperature of the water bath. This procedure is iterated until the highest target
temperature is reached then the entire experiment is repeated to check for data
consistency.

Step-by-step equipment operating instructions are provided in the notes to
instructors and students, which are included in the supporting information.

225 The selection of acetone as the test fluid was made with some care. An important
criterion was that even students with limited experimental skills should be able to
perform the experiment safely and within a reasonable time period, while producing
verifiably accurate results. The non-toxic nature of acetone minimises the risk when
conducting the experiment in case of poor manual handling skills. Moreover, its
230 relatively high volatility ensures that students obtain reliable results within a
reasonable timeframe, while working at a temperature range (25 to 50°C) that is well
below temperatures that would involve the danger of burns and scalds; for example,
acetone is more volatile than other safe-to-handle liquids such as acetonitrile, ethanol

and octane. A lesser, though not insignificant consideration, was that acetone
235 represents a reasonably economical choice of test fluid.

Students find that the most difficult part of the procedure is the adjustment of
the liquid levels in the isoteniscope by either venting the vacuumed ballast tank to
atmosphere or to the vacuum pump. When the pressure inside the ballast tank
increases too quickly, some of the liquid in the U-tube of the isoteniscope flows back
240 into the bulb together with the mixture of air and vapour of test liquid in the ballast
tank. It is then necessary to repeat the removal of air by vacuum, as described earlier in
this section. The necessary degree of care in manual handling and applying fine motor
skills in response to visual observation is an important learning experience.

245 HAZARD AND SAFETY PRECAUTIONS

Although acetone and *n*-hexane are chosen for their non-toxic nature, they are
still hazardous when inhaled or ingested. In addition, they are irritants to skin and
eyes. Therefore, personal protective equipment (PPE) including lab coat, safety goggles
and disposable gloves should be worn during the experiment in order to prevent direct
250 contact with the test fluids. Both acetone and *n*-hexane are highly flammable and hence
must be handled and stored away from sources of heat and ignition.

Another hazard is the possible implosion of glassware caused by the vacuum
created by the pump. It is important to tape the heavy-walled glassware in cling film to
contain potential projectiles in case of implosion. Before setting up the experimental
255 system, it is necessary to inspect the glassware for scratches, cracks and chips. The
experiment should be conducted in a well-ventilated area.

Risk assessments for the experiment and the use of hazardous chemicals are
also included in the supporting information.

260 RESULTS AND DISCUSSION

Vapour-Pressure Measurements for Acetone

To illustrate the effectiveness of the experiment, and the level of accuracy that may be expected, we report the results of 20 randomly chosen experiments carried out by first-year undergraduate students, each within an allotted time of three hours, using acetone. The measured vapour pressures are presented in Figure 4 together, for comparison, with a curve obtained with the Antoine equation^{14,15} (see also Table 1), representing the accepted true vapour pressure curve. Even though the students were relatively inexperienced in performing experiments, the data points fit closely to the Antoine curve (the coefficient of determination, $R^2 = 0.995$). This demonstrates both the efficacy of the experiment and the reproducibility of the measurements.

Figure 4. Vapour pressures of acetone as measured by undergraduate students together, for comparison, with the vapour-pressure curve generated using the Antoine equation (see Table 1).

275 Table 1: Coefficients in the Antoine equation: $\log_{10}(p_{\text{sat}}) = A - \frac{B}{T + C}$

Substance	Temperature range / K	A	B	C	Reference
Acetone	259.16–507.60	4.42448	1312.253	– 32.445	NIST database, using data from Ambrose <i>et al.</i> (1974) ¹⁶
<i>n</i> -Hexane	286.18–342.69	4.00266	1171.53	– 48.784	Williamham <i>et al.</i> (1945) (taken from NIST database) ¹⁷

Note: p_{sat} in bar and T in K.

Implementation in Teaching

Undergraduate students studying Chemical Engineering at Imperial College
 280 typically carry out the vapour-pressure experiment in the second term of their first year; every year, over 120 such students conduct the experiment, in pairs, in one three-hour laboratory session. For logistical reasons, many of the students perform the experiment before they have been introduced to the Gibbs function or the concept of chemical potential in their Thermodynamics lecture course, whereby they are also unfamiliar
 285 with the Clausius-Clapeyron equation and its application for the calculation of molar heats of vaporisation. Accordingly, they are supplied with a laboratory script designed on the basis of the students' background knowledge of the subject area to bridge the knowledge gap between experimental objectives and existing understanding. The laboratory script provides a brief introduction to the key concepts of phase equilibrium.
 290 The only prerequisites are that the students understand that the heat, Q , related to a constant-pressure process is given by the enthalpy change, and that for a reversible process the Second Law provides that $\Delta S = Q/T$, where S is the entropy, and T the (absolute) temperature. The prerequisite is typically taught in Key Stage-5 of the UK school education system or equivalent levels of secondary education elsewhere. We find
 295 that students across a wide range of ability comfortably complete the experiment within the allotted time, producing good-quality measurements (as illustrated in Figure 4); this

is a testament to the successful design and operational simplicity of the experiment. The course module in which this experiment is taught usually receives well over two thirds student satisfaction. From student and demonstrator feedback we know that
300 students find the experiment to be a positive and rewarding learning experience.

Extension 1: Vapour Pressure Measurements for other Volatile Liquids and Binary Liquid Mixtures

305 This vapour-pressure experiment can be easily extended to binary liquid mixtures. Although we have not included such an extension in our undergraduate laboratory exercise, for illustration we have employed our apparatus to study the azeotrope of acetone / *n*-hexane, which occurs at a molar composition of 0.64/0.36.

Before considering the binary mixture, we first examine the other constituent
310 pure component, *n*-hexane. In Figure 5 we show the measured vapour pressures of *n*-hexane, again (for comparison) with reference to accepted values; in this case, we take pseudo-experimental values obtained from the NIST Chemistry Web-book, which are conveniently available for *n*-hexane. Similar to the case of acetone, the data measured using our teaching laboratory experimental set-up is in close agreement with the
315 accepted values. For validating the robustness of data, actual values of the vapour pressure obtained from NIST, in addition to its Antoine parameter fit, was also compared with the vapour pressure values measured at all relevant temperatures. The standard deviation between the values was found to be less or equal to ± 3 Torr.

Figure 5. Vapour pressures of *n*-hexane (connected filled-squares) measured using our teaching-laboratory apparatus. Note that the error bars in the horizontal direction are smaller than the size of the symbols. Also shown (for comparison) are accepted vapour pressures (continuous curve) from the NIST Chemistry Web Book¹⁸.

Although acetone is more volatile than *n*-hexane, the molar heats of vaporisation for both test liquids are close to identical, both are 31 kJ mol⁻¹. This is calculated based on the Clausius-Clapeyron equation by plotting $\ln p$ against $1/T$ (i.e. constructing a van't Hoff plot as shown in Figure 6) and then multiplying the gradient of the best-fit line with the gas constant. They are identical to literature values¹¹.

Figure 6. van't Hoff plots for (a) acetone and (b) *n*-hexane.

Through the van't Hoff plots, advanced students could also determine the molar entropy of vaporization (ΔS_m^{vap}) for the test compound from the y-intercept of the best-fit line,

370 which is equal to $\Delta S_m^{vap} / R$. The molar entropies of vaporization for acetone and *n*-hexane are $110 \text{ J mol}^{-1} \text{ K}^{-1}$ and $107 \text{ J mol}^{-1} \text{ K}^{-1}$ respectively.

In Figure 7 we present the measured vapour pressures of an acetone/*n*-hexane binary mixture at the azeotropic molar composition (0.64/0.36), and compare these
375 with reference data for an ideal mixture.

Figure 7. Vapour pressures of the acetone/*n*-hexane azeotrope *n*-hexane (connected filled-squares) measured using our teaching-laboratory apparatus. Note that the error bars in both horizontal and vertical directions are smaller than the size

of the symbols. Also shown (for comparison) are calculated vapour pressures (continuous curve) obtained using the
380 Antoine equations relating to the pure compounds (see Table 1).

As is evident from Figure 7, the measured vapour pressures of this azeotrope deviate significantly from the calculated values that would correspond to ideal mixture behaviour. This positive non-ideality means that the molecules of these two liquids have
385 lower affinity with one another than with other molecules of their own species, making the liquid mixture more volatile than either liquid in its pure form. The calculated average molar heat of vaporisation (30 kJ mol^{-1}) is similar to the value for the pure compounds, as expected.

Extension 2: Calculation of Activity Coefficients for Non-Ideal Mixtures

390 This experiment can also be used to exemplify the concept of the activity coefficient, as students can calculate the activity coefficients of the two components in the azeotrope to account for the non-ideal behaviour of a mixture.

395 Assuming non-ideal behaviour in the liquid phase, the equations are:

$$p_a = \gamma_a \cdot x_a \cdot p_a^* \quad (2)$$

$$p_b = \gamma_b \cdot x_b \cdot p_b^* \quad (3)$$

$$p_{mixture} = p_a + p_b = \gamma_a \cdot x_a \cdot p_a^* + \gamma_b \cdot x_b \cdot p_b^* \quad (4)$$

Here p_i is the partial pressure of component i in the mixture, p_i^* is the vapour
400 pressure of pure component i , γ_i is the activity coefficient of component i and x_i is its mole fraction in the liquid phase. Indices a , b and $mixture$ denote compounds a , b and their mixture, respectively.

In the vapour phase we assume ideal behaviour depending on the mole fraction y as follows:

405
$$p_a = y_a \cdot p_{mixture} \quad (5)$$

$$p_b = y_b \cdot p_{mixture} \quad (6)$$

In an azeotropic mixture, the mole fraction of a compound is the same in both phases:

$$x_a = y_a \quad (7)$$

410
$$x_b = y_b \quad (8)$$

By rearranging the equations, the activity coefficient is simply the ratio of the vapour pressure of the mixture and the vapour pressure of the pure component at a particular temperature:

$$\gamma_a = \frac{p_{mixture}}{p_a^*} = \frac{p_{mixture}}{10^{A_a - \frac{B_a}{T+C_a}}} \quad (9)$$

415
$$\gamma_b = \frac{p_{mixture}}{p_b^*} = \frac{p_{mixture}}{10^{A_b - \frac{B_b}{T+C_b}}} \quad (10)$$

At any temperature, non-ideal behaviour of an azeotrope can now be accounted for, and the vapour pressure can be predicted using the Antoine equations for the pure compounds in combination with equation (4):

420
$$p_{mixture} = \gamma_a \cdot x_a \cdot 10^{A_a - \frac{B_a}{T+C_a}} + \gamma_b \cdot x_b \cdot 10^{A_b - \frac{B_b}{T+C_b}} \quad (11)$$

For the acetone-*n*-hexane system, the activity coefficients were found to be γ_a =1.25, and γ_b =1.89 respectively.

As a final comment, we note that as an additional learning opportunity one may
425 draw students' attention to the similarity in the mathematical forms of the Clausius-
Clapeyron equation and the Antoine equation, which has been exploited in our current
work to represent accepted values of the pure-component vapour pressures. Indeed,
one can consider the Antoine equation to be a simple modification of the Clausius-
Clapeyron equation¹⁵, albeit that the parameters employed are generally empirical. It is
430 important that the Antoine equation is not used outside its stated temperature range of
validity; failing to observe this restriction can lead to absurd results due to the
empirical nature of the equation. This could form the basis of an interesting discussion
for more experienced students. However, for inexperienced first-year students such as
those in our own teaching laboratory, we recommend the more-focused, simpler
435 analysis based purely on the Clausius-Clapeyron equation, in which the constituent
quantities retain clear, physical meaning.

CONCLUSION AND OUTLOOK

The use of an isoteniscope represents a convenient, simple and effective “hands-
440 on” experiment in which the vapour pressure of a fluid may be measured. Students can
easily perform the experiment twice for one test fluid in three hours. The vapour-
pressure measurements are highly reproducible and, for the test fluids investigated
here (acetone and *n*-hexane), seen to be in good agreement with literature values. Data
obtained in this experiment can be used to calculate the molar heat of vaporisation
445 based on the Clausius-Clapeyron equation, again yielding values that compare
favourably with literature values. The experiment can be extended further to study
binary liquid mixtures, as illustrated for the case of acetone/*n*-hexane, to introduce the
concept of azeotropes and ideal / non-ideal binary mixtures to students.

ASSOCIATED CONTENT

Supporting Information

Notes to students and instructors and a detailed technical drawing of the isoteniscope are included in the supporting information section.

ACKNOWLEDGEMENTS

The essential contributions of Nicola Guirguis to the development of this experiment are very gratefully acknowledged. The authors also wish to acknowledge GPE Scientific Ltd for providing the technical drawing of the isoteniscope.

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